इंटरनेट मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

"जानने का अधिकार, जीने का अधिकार"
Mazdoor Kisan Shakti Sangathan
"The Right to Information, The Right to Live"

"पुराने को छोड नये के तरफ"
Jawaharlal Nehru
"Step Out From the Old to the New"

IS 1493-3 (1987): Methods of chemical analysis of iron ores, Part 3: Determination of titanium, chromium, vanadium, calcium and magnesium by atomic absorption spectrophotometry [MTD 13: Ores and Raw Materials]

"ज्ञान से एक नये भारत का निर्माण"
Satyanarayan Gangaram Pitroda
"Invent a New India Using Knowledge"

Act Number 22 of 2005
The Indian Parliament
RIGHT TO INFORMATION

Made Available By
Public.Resource.Org

"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

BLANK PAGE

BRUTUM FULMEN

IGNORANTIA JURIS NON EXCUSAT

PROTECTED BY COPYRIGHT

IS : 1493 (Part 3) - 1987
(Reaffirmed 2001)

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF IRON ORES

PART 3 DETERMINATION OF TITANIUM, CHROMIUM, VANADIUM, CALCIUM AND MAGNESIUM BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

(First Reprint JULY 1989)

UDC 622.341.1:543.422

© *Copyright* 1988

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 2

May 1988

IS : 1493 (Part 3) - 1987

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF IRON ORES

PART 3 DETERMINATION OF TITANIUM, CHROMIUM, VANADIUM, CALCIUM AND MAGNESIUM BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

Methods of Chemical Analysis of Ores, Minerals and Refractories Sectional Committee, SMDC 35

Chairman	*Representing*
Dr L. P. Pandey	National Metallurgical Laboratory (CSIR), Jamshedpur
Members	
Dr V. M. Chowdhury (*Alternate* to Dr L. P. Pandey)	
Shri J. Banerjee	Steel Authority of India Ltd (Durgapur Steel Plant), Durgapur
Shri A. C. Mallick (*Alternate*)	
Shri S. V. Bhagwat	Khandelwal Ferro Alloys Ltd, Khandelwal Nagar
Shri D. N. Gupta (*Alternate*)	
Shri P. Bhattacharya	Belpahar Refractories Ltd, Belpahar
Shri P. V. Rao (*Alternate*)	
Shri P. K. Ghosal	Kumardhubi Fireclay & Silica Works Ltd, Kumardbhubi
Shri L. N. Banerjee (*Alternate*)	
Shri N. B. Gude	Chowgule & Co Pvt Ltd, Goa
Shri S. S. Honavar	Italab Pvt Ltd, Madras
Shri M. V. Dahholkar (*Alternate* I)	
Shri J. C. Dey (*Alternate* II)	
Shri R. N. Misra	Hindustan Copper Ltd, Jhunjhunu
Shri D. C. Mathur (*Alternate*)	
Shri K. C. Modi	Hindustan Aluminium Corporation Ltd, Mirzapur
Shri B. K. Singh (*Alternate*)	
Shri V. T. Moorty	Tamil Nadu Fluorine & Allied Chemicals, Cuddalore
Shri V. S. Raghavan (*Alternate*)	
Shri M. Rae	National Test House, Calcutta
Shri A. K. Dutta Gupta (*Alternate*)	

(*Continued on page* 2)

© *Copyright* 1988
BUREAU OF INDIAN STANDARDS
This publication is protected under the *Indian Copyright Act* (XIV of 1957) and reproduction in whole or in part by any means except with written permission of the publisher shall be deemed to be an infringement of copyright under the said Act.

(*Continued from page* 1)

Members	*Representing*
DR J. RAJARAM	Essen & Co, Bangalore
SHRI K. RAMA KRISHNA (*Alternate*)	
SHRI G. RAJA RAO	Ferro Alloys Corporation Ltd, Shreeramnagar
SHRI D. V. RAMAN	National Mineral Development Corporation Ltd, Hyderabad
SHRI N. VENKATRAMAN (*Alternate*)	
SHRI V. K. RAO	Mineral Exploration Corporation Ltd, Nagpur
SHRI H. N. SAHA	Steel Authority of India Ltd (Bhilai Steel Plant), Bhilai
SHRI M. ROY CHOWDHARY (*Alternate*)	
SHRI M. R. SAHA	Central Glass & Ceramics Research Institute (CGCRI), Calcutta
SHRI N. C. SAHA	Harry Refractories & Ceramic Works Pvt Ltd, Calcutta
DR N. R. SENGUPTA	Geological Survey of India, Calcutta
DR A. K. DAS (*Alternate*)	
DR R. C. SHARMA	Rajasthan State Mineral Development Corporation Ltd, Jaipur
DR Y. P. SRIVASTAVA	Tata Iron & Steel Co Ltd, Jamshedpur
DR K. S. R. KRISHNAIAH (*Alternate*)	
SHRI B. N. TIKOO	Department of Atomic Energy, Hyderabad
DR CH. VENKATESWARLU	Bhabha Atomic Research Centre, Bombay
SHRI B. MUKHERJI, Director (Struc & Met)	Director General, BIS (*Ex-officio Member*)

Secretary

SHRI M. L. SHARMA
Assistant Director (Metals), BIS

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF IRON ORES

PART 3 DETERMINATION OF TITANIUM, CHROMIUM, VANADIUM, CALCIUM AND MAGNESIUM BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

0. FOREWORD

0.1 This Indian Standard (Part 3) was adopted by the Bureau of Indian Standards on 24 August 1987, after the draft finalized by the Methods of Chemical Analysis of Ores, Minerals and Refractories Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 IS : 1493 (Part 1)-1981* covers the chemical analysis of iron ores for determination of moisture, silica, iron, phosphorus ($\geqslant$0·5 percent), aluminium and sulphur by conventional methods, namely gravimetric and volumetric method. The chemical analysis for determination of remaining constituents given in IS : 1493-1959† will be covered in Part 2 of this standard which is under preparation. With the publication of IS : 1493 (Part 2), IS : 1493-1959† will be withdrawn.

0.3 Since atomic absorption spectrophotometry (AAS) for iron ore has greatly facilitated the determination of many of the constituents and is adopted as a preferred procedure for many trace elements, a need was felt to publish a standard for determination of various constituents in iron ores by atomic absorption spectrophotometry.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960‡.

1. SCOPE

1.1 This standard (Part 3) prescribes the atomic absorption spectrophotometric method for determination of titanium, chromium,

---

*Methods of chemical analysis of iron ores: Part 1 Determination of common constituents (*first revision*).

†Methods of chemical analysis of iron ores.

‡Rules for rounding off numerical values (*revised*).

vanadium, calcium and magnesium in iron ores. The method is applicable to a concentration range of 0·05 to 5 percent of titanium dioxide, chromium oxide, vanadium pentoxide and 0·01 to 10 percent of oxides of calcium and magnesium.

2. SAMPLING

2.1 Samples for determination shall be drawn and prepared in accordance with IS : 1405-1982*.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, analytical grade reagents and distilled water (*see* IS : 1070-1977†) shall be employed in the test.

4. GENERAL

4.1 Use of Filter Papers — In the methods prescribed in this standard, relative number of Whatman filter paper only have been prescribed, since these are commonly used. However, filter paper of any other suitable brand with equivalent porosity may be used.

5. OUTLINE OF THE METHOD

5.1 The test portion is decomposed by heating with hydrochloric acid. Insoluble residue is hydrofluorised, fused with sodium carbonate and taken up with the main solution. Individual elements are determined by AAS after proper dilutions and addition of background solutions.

6. REAGENTS

6.1 Concentrated Hydrochloric Acid — rd = 1·16 conforming to IS : 265-1976‡.

6.2 Concentrated Nitric Acid — rd = 1·42 conforming to IS : 264-1976§.

6.3 Dilute Hydrochloric Acid — 1 : 10 (*v/v*).

6.4 Dilute Sulphuric Acid — 1 : 1 (*v/v*) and 2 N.

6.5 Hydrofluoric Acid — 48 percent.

6.6 Sodium Carbonate (Anhydrous) — Solid.

---

*Methods of sampling iron ores (*second revision*).

†Specification for water for general laboratory use (*second revision*).

‡Specification for hydrochloric acid (*second revision*).

§Specification for nitric acid (*second revision*).

6.7 Background Solution A — Dissolve 15·0 g of metallic iron (iron wire 99·9 percent purity) in 100 ml concentrated hydrochloric acid and 5 ml of concentrated nitric acid, dilute to one litre.

6.8 Background Solution B — Dissolve 75 g of tartaric acid and 35 g of potassium chloride in a mixture of 50 ml concentrated hydrochloric acid and 200 ml water. Add 10 ml of dilute sulphuric acid (1:1) and dilute to 500 ml.

6.9 Standard Titanium Solution (1 000 μg TiO_2/ml) — Fuse 0·500 g of titanium dioxide (previously dried at 110°C) with 5 g of potassium pyrosulphate in a silica crucible. Dissolve the cold melt in 100 ml of warm dilute sulphuric acid (2 N). Transfer to a 500 ml volumetric flask and make up volume with dilute sulphuric acid (2 N).

6.10 Standard Chromium Solution (1 000 μg Cr_2O_3/ml) — Dissolve 1·930 g of potassium dichromate (previously dried at 110°C) in one litre water in a volumetric flask.

6.11 Standard Vanadium Solution (1 000 μg V_2O_5/ml) — Dissolve 1·000 g of vanadium pentoxide (previously dried at 110°C) in 20 ml dilute sodium hydroxide solution (5 percent). Add about 100 ml water and 10 ml of dilute sulphuric acid (1:1), transfer to a one litre volumetric flask and make up volume with water.

6.12 Standard Calcium Solution (100 μg CaO/ml) — Dissolve 0·178 g of calcium carbonate (previous dried at 110°C) in 20 ml of dilute hydrochloric acid (4 N). Transfer to a one litre volumetric flask and make up volume with water.

6.13 Standard Magnesium Solution (100 μg MgO/ml) — Dissolve 0·600 g of magnesium metal (magnesium wire, 99·99 percent purity) in 20 ml of concentrated hydrochloric acid. Transfer to one litre volumetric flask and make up volume.

Take 10 ml of above solution to 100-ml volumetric flask and make up volume with water.

6.14 Mixed Calibration Solution for Titanium, Chromium and Vanadium — Prepare 0, 5, 10, 20, 30, 40 and 50 μg/ml mixed standards by taking, 0, 2·5, 5, 10, 15, 20 and 25 ml of standard titanium solution, standard chromium solution, standard vanadium solution in 500-ml volumetric flasks. Add 50 ml of background solution A and 50 ml background solution B to each flask, dilute to volume and mix well.

6.15 Mixed Calibration Solutions for Calcium and Magnesium — Prepare 0, 0·5, 1·0, 2·0, 4·0, 6·0, 8·0 and 10·0 μg/ml mixed standards by taking 0, 2·5, 5, 10, 20, 30, 40 and 50 ml of standard calcium solution and standard magnesium solution in 500-ml volumetric flasks. Add 50 ml of background solution A and 50 ml of background solution B to each flask, dilute to volume and mix well.

7. APPARATUS

7.1 Atomic absorption spectrophotometer fitted with nitrous oxide — acetylene burner head. Instrument parameters may vary with the instrument and the manufacturer's instructions may be followed in each case.

8. PROCEDURE

8.0 The analysis shall be carried out generally in duplicate, independently on each sample. In each run, one analysis of a standard sample of the same type of ore and one blank test shall be carried out in parallel with the analysis of test samples.

8.1 Preparation of Sample Solution — Weigh 2·500 g sample in a 500-ml beaker. Moisten it with a few ml of water, add 75 ml of concentrated hydrochloric acid, cover with a watch glass and heat to gentle boiling until the sample is nearly decomposed and no further attack is visible. Remove the cover glass, add 5 ml of concentrated nitric acid and continue heating for 15 minutes more. Cool, add about 100 ml of water and filter through a Whatman No. 41 filter paper. Wash 4-5 times with dilute hydrochloric acid and finally with hot water until the filter paper is colourless. Collect the filtrate and washings in a 250-ml volumetric flask (main solution).

8.1.1 Ignite the residue with the filter paper in a platinum crucible at about 900°C. Cool the crucible, moisten the ignited mass with a few drops of water, add 2 drops of dilute sulphuric acid (1:1) and about 10 ml of hydroflouric acid. Place the crucible on a hot plate and continue heating until the mass becomes dry. Remove the crucible from the hot plate and ignite it at about 900°C for few minutes. Cool, add 0·5 g of sodium carbonate and fuse the mass for 15 minutes over a burner. Cool, dissolve the fused mass in about 20 ml dilute sulphuric acid (2 N) solution and transfer the solution and add to the main filtrate preserved in 250-ml volumetric flask. Make up volume with water and mix well.

8.2 Preparation of Test Solutions for Atomic Absorption Measurements — Prepare test solutions for AAS measurements as per Table 1.

TABLE 1 DILUTION GUIDE FOR AAS MEASUREMENTS

(*Clause* 8.2)

TEST SOLUTION No.	ANALYTE ELEMENT OXIDE	CONCENTRATION RANGE Percent	ALIQUOT OF MAIN SOLUTION (8.1) TAKEN ml	BACK-GROUND SOLUTION A (6.7) ADDED ml	BACK-GROUND SOLUTION B (6.8) ADDED ml	FINAL VOLUME ml
1.	Ti, V, Cr	0·05-1	50	Nil	10	100
2.	Ti, V, Cr	1-5	10	5	10	100
	Ca, Mg	0·01-1				
3.	Ca, Mg	1-10	5	50	50	500

8.3 Atomic Absorption Measurements

8.3.1 Adjust the atomic absorption instrument using instrument parameters recommended by the manufacturer. Use nitrous oxide acetylene flame for all the elements. During measurements of magnesium, the burner head should be rotated through an angle of 90° to the light path, that is, the burner head should be perpendicular to the light path.

8.3.2 Aspirate water to establish that the absorbance reading is not drifting and then set the initial reading for water to zero absorbance. Aspirate the calibration solutions (**6.14** or **6.15**) and the test solutions (*see* Table 1) in the order of increasing absorption, starting with the blank solution and the zero calibration solution. When a stable response has been obtained for each solution, record the readings. Aspirate water between each calibration solution and test solution. Repeat the measurements two times more.

8.3.3 Convert the average of the readings for each calibration solution to absorbance. Obtain the net absorbance of each calibration solution by subtracting the average absorbance of the zero calibration solution. In a similar manner, obtain the net absorbance of the test solution by subtracting the absorbance of the blank solution. Prepare calibration curve by plotting the net absorbance values of the calibration solutions against microgram of analyte per millilitre. Convert the net absorbance values of the test solution to microgram of analyte per millilitre by means of the calibration curve.

8.4 Calculation — The analyte concentration, as percentage of mass is calculated from the following relation:

$$M, \text{ percent} = \frac{T}{W} \times 100$$

where

M = analyte of element oxide,

T = microgram of analyte element per millilitre of test solution obtained from calibration curve, and

W = sample concentration in µg/ml in the test solution.

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones: 331 01 31, 331 13 75 Telegrams: Manaksanstha
(Common to all Offices)

Regional Offices:

		Telephone
Central	Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	331 01 31 331 13 75
*Eastern	1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola, CALCUTTA 700054	36 24 99
Northern	SCO 445-446, Sector 35-C, CHANDIGARH 160036	2 18 43 3 16 41
Southern	C. I. T. Campus, MADRAS 600113	41 24 42 41 25 19 41 29 16
†Western	Manakalaya, E9 MIDC, Marol, Andheri (East), BOMBAY 400093	6 32 92 95

Branch Offices:

'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMADABAD 380001	2 63 48 2 63 49
‡Peenya Industrial Area 1st Stage, Bangalore Tumkur Road BANGALORE 560058	38 49 55 38 49 56
Gangotri Complex, 5th Floor, Bhadbhada Road, T. T. Nagar, BHOPAL 462003	6 67 16
Plot No. 82/83, Lewis Road, BHUBANESHWAR 751002	5 36 27
53/5, Ward No. 29, R.G. Barua Road, 5th Byelane, GUWAHATI 781003	3 31 77
5-8-56C L. N. Gupta Marg (Nampally Station Road), HYDERABAD 500001	23 10 83
R14 Yudhister Marg, C Scheme, JAIPUR 302005	6 34 71 6 98 32
117/418 B Sarvodaya Nagar, KANPUR 208005	21 68 76 21 82 92
Patliputra Industrial Estate, PATNA 800013	6 23 05
T.C. No. 14/1421, University P.O., Palayam TRIVANDRUM 695035	6 21 04 6 21 17

Inspection Offices (With Sale Point):

Pushpanjali, First Floor, 205-A West High Court Road, Shankar Nagar Square, NAGPUR 440010	2 51 71
Institution of Engineers (India) Building, 1332 Shivaji Nagar, PUNE 411005	5 24 35

---

*Sales Office in Calcutta is at 5 Chowringhee Approach, P. O. Princep Street, Calcutta 700072 27 68 00

†Sales Office in Bombay is at Novelty Chambers, Grant Road, Bombay 400007 89 65 28

‡Sales Office in Bangalore is at Unity Building, Narasimharaja Square, Bangalore 560002 22 36 71

Reprography Unit, BIS, New Delhi, India